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UTILITY PATENT APPLICATION TRANSMITTAL

(Only for new nonprovisional applications under 37 CFR 1.53(b))

Attorney Docket No. AD6552USNA1

First Named Inventor or Application Identifier

STEPHEN P. SCHEINBERG

Express Mail Label No. EK219471143

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APPLICATION ELEMENTS

See MPEP chapter 600 concerning utility patent application contents.

ADDRESS TO:

Assistant Commissioner for Patents
Box Patent Application
Washington, DC 20231

1. ☒ Fee (Authority to charge deposit account below.)
(Submit an original, and a duplicate for fee processing)
2. ☒ Specification [Total Pages 17]
(preferred arrangement set forth below)
- Descriptive title of the invention
 - Cross References to Related Applications (if needed)
 - Statement Regarding Fed sponsored R & D (if needed)
 - Reference to Microfiche Appendix (if filed)
 - Background of the Invention
 - Brief Summary of the Invention
 - Brief Description of the Drawings (if filed)
 - Detailed Description
 - Claim(s)
 - Abstract of the Disclosure
3. ☒ Drawing(s) (35 USC 113) [Total Sheets 4]
4. ☒ Oath or Declaration [Total Pages 1]
- a. ☐ Newly executed (original or copy)
 - b. ☒ Copy from a prior application (37 CFR 1.63(d))
(for continuation/divisional with Box 14 completed)
 - i. ☐ **DELETION OF INVENTORS**
Signed Statement below at 15 deleting inventor(s) named in the prior application, see 37 CFR 1.63(d)(2) and 1.33(b).
5. ☒ Incorporation by Reference (useable if Box 4b is checked)
The entire disclosure of the prior application, from which a copy of the oath or declaration is supplied under Box 4b, is considered as being part of the disclosure of the accompanying application and is hereby incorporated by reference therein.

6. ☐ Microfiche Computer Program (Appendix)
7. ☐ Nucleotide and/or Amino Acid Sequence Submission
(if applicable, all necessary)
- a. ☐ Computer Readable Copy
 - b. ☐ Paper Copy (identical to computer copy)
 - c. ☐ Statement verifying identity of above copies

ACCOMPANYING APPLICATION PARTS

8. ☐ Power of Attorney
9. ☐ Information Disclosure Statement (IDS)/Cover Letter plus PTO-1449 ☐ Copies of IDS Citations
10. ☐ Preliminary Amendment
11. ☒ Return Receipt Postcard (MPEP 503)
(Should be specifically itemized)
12. ☐ Certified Copy of Priority Document(s)
(if foreign priority is claimed)
13. ☐ Other:

14. If a CONTINUING APPLICATION, check appropriate box and supply the requisite information:

☐ Continuation ☒ Divisional ☐ Continuation-in-part (CIP) of prior Application No.: 09/054,771

15. ☐ **DELETION OF INVENTOR(S) STATEMENT:** This application is being filed by less than all the inventors named in the prior application. In accordance with 37 CFR 1.63(d)(2) and 1.33(b), the Assistant Commissioner is requested to delete the name(s) of the following person or persons who are not inventors of the invention being claimed in this application:
16. ☒ Amend the specification by inserting before the first line the sentence:
-- This is a division of Application No. 09/054,771 filed April 3, 1998, now pending. --
17. ☒ Cancel in this application original claims 1-35 of the prior application before calculating the filing. (At least one original independent claim must be retained for filing purposes.)
18. ☐ Priority of foreign Application No. _____ filed on _____ in _____
_____ is claimed under 35 U.S.C. 119.

| CLAIMS | (1) FOR | (2) NUMBER FILED | (3) NUMBER EXTRA | (4) RATE | (5) CALCULATIONS |
|--------|--|------------------|------------------|--------------------------------------|------------------|
| | TOTAL CLAIMS (37 CFR 1.16(c)) | 7 - 20 = | 0 | x \$ 18 = | 0 |
| | INDEPENDENT CLAIMS (37 CFR 1.16(b)) | 3 - 3 = | 0 | x \$ 78 = | 0 |
| | MULTIPLE DEPENDENT CLAIM(S) (if applicable) | | | + \$ 260 = | 0 |
| | | | | BASIC FEE (37 CFR 1.16(a)) | + \$ 690.00 |
| | | | | TOTAL | \$ 690.00 |

19. The Commissioner is hereby authorized to credit overpayments or charge the following fees to Deposit Account No. 04-1928:

a. ☒ Fees required under 37 CFR 1.16.

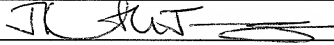
b. ☒ Fees required under 37 CFR 1.17.

20. ☐ Other:

21. CORRESPONDENCE ADDRESS

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22. SIGNATURE OF ATTORNEY OR AGENT REQUIRED

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| NAME | J. Kenneth Joung | REG. NO.: 41,881 |
| SIGNATURE |  | |
| DATE | April 25, 2000 | |

WETLAY PROCESS FOR MANUFACTURE OF HIGHLY-ORIENTED FIBROUS MATS

GOVERNMENT INTEREST

- 5 The invention described herein was made in the course of work under a grant or award from National Institute of Standards and Technology (NIST).

BACKGROUND OF THE INVENTION

Field of the Invention

- 10 The present invention is directed generally to processes for forming layers of fibrous material and, more specifically, to a wetlay process for manufacturing highly-oriented fibrous mats.

Description of the Related Art

- 15 Wetlay processes for manufacturing fibrous mats have typically been directed to the use of long glass, mineral wool or carbon fibers on both inclined wire wetlay machines and on rotary formers (cylinder machines). Typical wetlay processes involve injecting stock containing a plurality of fibers into the headbox of a wetlay machine. Suction under a wirebelt draws fibers within the stock toward the wirebelt to ultimately form a fibrous mat. In general, fiber orientation
- 20 is often controlled to make it as random (square or 1:1 strength profile) as possible. Various existing patents depict machinery improvements to prevent shear boundary layers which might tend to form small areas of oriented fiber. For example, such shear boundary layers often form at the side walls of the headbox or between adjacent stock flows into the headbox. This is because inadvertent
- 25 fiber alignment in the machine direction reduces transverse (cross machine) mat strength.

- Typical glass mat machines may produce a maximum of 1.4 to 1 machine direction (MD) to cross-machine direction (CD) orientation (58% MD orientation), because the suction (forming) wire speed is higher than the incoming
- 30 water speed. A few machines have been known to orient at a 4 to 1 ratio (80%), while even fewer machines have been known to orient at a 6 to 1 ratio (6/7 = 85.7 %).

In general, degree of orientation is measured as:

$$[\text{MD} / \text{CD}] / [(\text{MD} / \text{CD}) + 1]$$

where the span between the jaws of the tensile tester is longer than the longest reinforcement fiber in the structure to avoid bridging the gap.

- 5 All prior attempts, however, have failed to produce a greater than 90% wetlay orientation (9 to 1 MD to CD strength ratio or greater). As such, there exists a need to develop fibrous mats having the strength characteristics associated with a mat having greater than 90% wetlay orientation. In addition, many prior attempts to improve existing machinery required the use of nozzles to increase
- 10 fiber velocity. Such prior attempts have not, however, readily lent themselves to retrofitting existing machinery. As such, there is currently a need to develop a cost-effective and efficient system to retrofit existing machinery so that they are capable of providing mats with at least a 90% wetlay orientation.

15 SUMMARY OF THE INVENTION

- In accordance with the present invention, the invention includes a method of producing highly-oriented fibrous mats having at least a 90% machine direction orientation including the steps of producing a thickened solution containing a plurality of suspended fibers, introducing the thickened suspension into a headbox
- 20 of a wetlay machine and decelerating the fiber suspension to a velocity less than wirebelt operating velocity, and applying suction through the wirebelt to orient and pin the fibers on the wirebelt.

- The present invention also includes a method of retrofitting an existing headbox of a wetlay machine so as to produce highly-oriented fibrous mats,
- 25 including the steps of increasing head level within the headbox to increase headbox stock capacity, and accelerating operating velocity of a wirebelt within the wetlay machine beyond an operating velocity of stock entering the headbox.

- The present invention also includes end products made of a plurality of mats, each of the mats including a plurality of discontinuous reinforcement fibers
- 30 having at least a 90% machine direction orientation.

BRIEF DESCRIPTION OF THE DRAWINGS

- Figure 1 is a schematic illustration of a wet-laying process used in the present invention.
- 35 Figure 2 is a view of an inclined wire wetlay machine incorporating features of the present invention.

Figure 2A is a blown-up portion of Figure 2.

Figure 3 is a view of a rotary cylinder wetlay machine incorporating features of the present invention.

Figure 3A is a view of a standard rotary cylinder which suffers from "dead" spots containing eddy current formations.

5 DETAILED DESCRIPTION OF THE PRESENT INVENTION

With reference to Figure 1, a wet laying process used in an embodiment of the present invention is shown. The process utilizes paper making equipment which may include a pulper 1, a transfer pump 2, an agitated supply tank 3, the headbox 4 of an inclined wire paper machine 5, a suction box 11, a dewatering section 6, and a windup or driven spool 7. In operation, reinforcement fibers and thermoplastic fibers are dispersed in water in pulper 1. The slurry is transferred via a pump 2 from the pulper to an agitated supply tank 3. Feed stock from the supply tank is then pumped to the headbox 4. Dilution water is added from tank 8 to the feed line before the headbox 4 to reduce stock consistency. The slurry is drained through the wire by suction box 11 and forms a mat 9 which is dewatered by passing over suction slots 6 in the dewatering section. The dewatered sheet is then wound in damp form on driven spool 7. The sheet 9 wound on the spool 7 is unwound in layers and dried. Alternatively, the dewatered sheet is passed through a convection oven, dried and/or fused, and wound-up.

20 With reference to Figures 2-3A, two embodiments of the present invention will now be shown and described in greater detail. In general, fibers in the present invention are aligned as they move toward a belt in a large open body of thickened fluid. The moving belt operates at a higher speed than the approaching water and fibers. A nozzle for pre-orienting the fibers by increasing fiber and fluid velocity is not needed.

25 With reference to the Figures, discontinuous reinforcement fibers are uniformly and individually dispersed in a thickened water containing a thickener and a wetting agent which are selected for compatibility with the solids to be dispersed and the chemistry of surface finishes supplied on the solids. Optionally, discontinuous thermoplastic fibers or particles may also be added to the thickened water. The discontinuous reinforcement fibers are typically 3/4" to 1.25" long (1.9 to 3.2cm). However, these discontinuous reinforcement fibers may be as long as 2.5" (6.4cm) or as short as 0.039 inches (1mm). Viscosity is typically set at 1.5 centipoise or greater, although it is to be understood that the viscosity may be set at other values. When shear thinning (thixotropic) thickening systems are used, viscosity is typically set at 8 centipoise or greater.

In one embodiment of the present invention, the reinforcement fibers are all one length, diameter, and material. In the alternative, the reinforcement fibers may have a distribution of lengths and/or diameters. The reinforcement fibers may also consist of a mixture of materials, stiffnesses, and percentage compositions. The reinforcement fibers may include but are not limited to: PAN (polyacrylonitrile) or Pitch based carbon (graphite), glass, para-aramid, ceramics, metals, high temperature thermoplastics, thermosets, liquid crystal polymer fibers, ultra high molecular weight polyethylene, natural fibers, natural or synthetic spiderweb. The reinforcement fibers may also have surface treatments or finishes designed to promote adhesion to a thermoplastic component. The reinforcement fiber may have a surface which is oxidized to promote water dispersion and adhesion. Surface oxidation of carbon fibers may be provided, for example, by ozone treatment. The surface modification of reinforcement fibers may also be provided by plasma treatment in selected species. It is to be noted that the preferred concentration of the reinforcement fiber component to the thermoplastic component is 60-70 weight % reinforcement fiber and 40-30 weight % thermoplastic component. Although either or both drawn and undrawn thermoplastic fibers may be used, undrawn fibers are preferred as drawn fibers may cause wrinkling/misalignment within the mat.

In other embodiments of the present invention, the thermoplastic component may be a fiber, granular particle or flat platelet, although the preferred form of thermoplastic component is fiber. The preferred fiber length falls in the range of 0.6 to 1.3 cm. (0.25 inch to 0.5 inch.) In other embodiments, the thermoplastic component is fibers of a single material and length, and/or one of mixed materials, forms, melting points, sizes(lengths & diameters), molecular weights, and/or mixture composition (%). The thermoplastic components may include, but are not limited to, polyethylene, polypropylene, polyethylene terephthalate (PET), polyamides, polyethylene naphthalate (PEN), polyetheretherketone (PEEK) and polyetherketoneketone (PEKK). The thermoplastic component may be cross-linkable in a later process step. The thermoplastic component may contain additives, including, but not limited to: fillers, antioxidants, color, electrically or thermally conductive or insulating materials, adhesion aids, melt flow modifiers, cross-linking agents, and chemically or biologically reactive materials, and molecular sieves.

In one embodiment of the present invention, an antifoaming agent is added to the thickened water to prevent entrainment of fibers which entangle in the floating foam, and reduce orientation.

Typically, prior to introduction to the headbox, stock is dispersed with a 0.5 to 2 weight % solids content and diluted to 0.05 % to 0.2% with thickened water of the same composition. In the alternative, the final dilution concentration may be mixed and pumped directly to the headbox. While dissimilar fibers may be added in any order, including simultaneously, it is preferred that thermoplastic fibers be dispersed before the reinforcement fibers to aid dispersion and reduce mixing time which may cause breakage damage to high modulus fibers. Alternatively, reinforcement fibers and thermoplastic fibers may be dispersed separately and then combined in a stock tank or in line to the headbox.

With reference to Figures 2 and 3, dispersed stock 10 is uniformly introduced across the width of an open headbox 20 of an inclined wire wetlay machine or an open headbox 30 of a rotary cylinder wetlay machine. Because the headbox is open, the surface of the water is open to atmospheric pressure. Stock flow in the headbox is designed to a) minimize turbulence and fiber entanglement, b) slow or stall fiber velocity, c) maintain individual fiber separation, and d) promote laminar flow of fibers toward the suction wire so that (1) out of plane (through direction) fiber deposition is minimized, (2) a thin flat mat is formed, and (3) translation of machine direction modulus (in subsequent applications such as consolidated structural sections) is increased.

In the inclined wire wetlay machine of Figure 2, stock entering the headbox flows substantially vertically, as shown at reference numeral 40, against a liquid head 50 which is maintained at a height greater than the highest vertical position of the last suction box 61 of a plurality of suction boxes 60 under the moving forming wire 70 by a regulator weir 80, the bottom edge of which is spaced sufficiently higher than the wire surface so as to not to interfere with the mat 85 as it exits, or to influence fiber orientation. The forming "wire" 70 is a porous moving belt typically made of woven metal wire or synthetic filaments. Preferably, the belt has a square or rectangular weave pattern. The belt may also be a woven, nonwoven, multilayer or knit fabric, or have a carrier fabric lying on the moving wire belt. Although the present invention may be used with a twill weave belt and successfully achieve a greater than 90% oriented mat, the twill weave belt will collect fibers in angled grooves between the wires, thereby reducing machine direction orientation.

With continuing reference to Figures 2 and 3, the stock stream must turn 60 to 180 degrees at reference point 90 in order to approach the forming wire. Fiber velocity is slowed substantially, turbulence is greatly reduced, and flow in the body of the stock stream approaching the suction wire becomes substantially

laminar at reference point **100**. A separate plate or extension **110** to the rear upper portion of the headbox may be added to deflect fibers under the surface to prevent floating and entanglement.

With reference to Figure 2, the linear velocity of the porous collecting surface **70** is set equal to or greater than 3 times the linear velocity of the stock in the body at point **90** in the body of the headbox (typically 4-8 times or more). Preferably, however, the ratio of linear wire velocity to velocity of water in the body of the headbox is between 4:1 and 10:1. Gravity or vacuum assisted suction boxes **60** aligned across the underside of the forming wire and spaced along its path, accelerate the aqueous dispersion locally, pull the liquid through the moving wire screen, and pin the fibers to the wire.

With reference to Figure 2A, a blown-up portion **115** of the suction boxes is shown. As the randomly oriented fiber dispersion **120** approaches the wire surface, the locally increased liquid velocity begins to rotate the fibers **125** so they partially orient at point **130** in the direction of the local flow streamline. The leading ends of the fibers **140** are pinned to the wire by suction. The higher velocity wire drags the fibers into alignment **150** as the rest of their lengths are pinned to the belt. Successive oriented layers of fiber are deposited as the wire moves across the suction boxes. Suction may be increased by vacuum assist to control fiber pinning along the length of the forming section. This is useful for maintaining orientation in the upper layers of heavier weight mat.

In one embodiment of the present invention, the stock enters the inclined wire headbox uniformly across its width, and substantially vertically upward against the liquid head thus slowing the fibers, and must turn essentially right angles proportionately to present the fibers to the wire with reduced turbulence (in a more laminar flow), and with reduced linear velocity. The open head of stock in the inclined wire machine may be set higher, typically 18 to 26 cm (7 - 10 inches) than the exit point of the last suction box **61** in the formation section. In another embodiment, stock entering the headbox is guided in a substantially backward and upward direction from the direction of belt motion, and must slow against the head, reverse direction in a smooth flow pattern, and present the fibers to the wire with reduced velocity and turbulence.

In the rotary cylinder wetlay machine of Figure 3, the headbox entry **160** directs the incoming stock upward and to the rear of the headbox (opposite to the exit direction). In the preferred embodiment, the rear of the headbox is streamlined to the natural hydraulic curvature **170** of the stock flow as it reverses direction and moves in a laminar flow **100** toward the forming wire **190** which is

supported on a rotating cylindrical drum 200 and is moving at 3 times or greater the linear velocity of the stock at point 90 in the headbox. Suction boxes 210 under the wire cause the reinforcement fibers to deposit with greater than 90% machine direction orientation by the same mechanism as described for the inclined wire machine.

With reference to Figure 3A, the streamlined rear headbox design of Figure 3 eliminates "dead" spots 220 in which eddy current formation causes fiber entanglement and reduces orientation. In one embodiment of the present invention, such a streamlined headbox conforms to the natural streamline flow of the stock.

It is also to be understood that a rotary former is a form of infinitely varying inclined wire machine.

With reference to Figures 2 and 3, the mat 85 formed has greater than 90% orientation and in the preferred form, greater than 95% machine direction orientation of reinforcement fiber. It is suitable for manufacture of strong, stiff composites with engineered properties. When it contains a thermoplastic component, it can be melted and stabilized in an in-line convection oven. When the mat contains a thermoplastic component, it is preferentially dried and bonded in a through-air convection oven, and wound on rolls. The mat may also be sprayed or saturated with chemical binder or size and dried in a continuous oven. The mat may also be dried and wound in rolls without binder. An interleaf layer may also be used. The typical areal or basis weight range is 68 to 339 gm./square meter (2 to 10 oz./square yard), (42 to 208 pounds per 3000 square foot ream), (0.014 to 0.069 pounds/square foot).

25 Test Results

I. In a first series of tests, a 12 inch (30.5cm) wide, open headbox inclined wire forming machine configured as in Figure 1 was used to produce 400 foot (12.2 meter) rolls of oriented mats of Glass/PET, Pan Carbon/PET, and Pitch Carbon/PET on a rectangular weave smooth top surface synthetic wire belt. All process water was thickened to 1.8 centipoise with polyacrylamide viscosity modifier at 0.5% concentration in the water. Surface active agent, and antifoam were added, and pH was adjusted to 8.0-8.2 with ammonia. The initial mix was, in each case, 0.5 % total fiber by weight, and the diluted stock entered the headbox at 0.17 % solids.

35 A regulator plate was used as a dam to increase hydrostatic head to 7-9 inches (18 to 23cm) above the height of the trailing edge of the last suction box. Total head above the leading edge of the first suction box on the inlet end of the

machine was maintained at 17-19 inches (43 to 48cm). The bottom of the regulator was spaced 0.5 inches (1.3cm) above the wire, and did not contribute to fiber orientation.

- For this series of tests, the mat was dried and heated without pressure in a muffler oven at 325 degrees Centigrade to melt the thermoplastic PET fibers. MD and CD tensile strength was measured on 3 inch (7.6cm) wide samples with a 3 inch (7.6cm) span.

Operating variables and resultant mat orientation ratios are:

| Identification: | A | B | C | D |
|---|------------------------|-------------------------|------------------------|-------------------------|
| Reinforcing Fiber | Glass | Glass | PAN Carbon | Pitch Carbon |
| Reinf. Fiber Modulus Million PSI (gigapascals GPa) | 10.5 (72.4) | 10.5 (72.4) | 33 (228) | 82 (565) |
| Wt % Reinf. Fiber Vol. % Reinf. Fiber | 60 44 | 70 52 | 60 54 | 60 49 |
| Length, inches (cm) Reinf. Fiber PET fiber | 1 (2.5) 0.5 (1.3) | 1.25 (3.2) 0.5 (1.3) | 1.0 (2.5) 0.5 (1.3) | 1.25 (3.2) 0.5 (1.3) |
| Velocity differential feet/minute (meters/minute) Stock Forming Wire | 25 (7.6) 100 (30.5) | 25 (7.6) 200 (61) | 25 (7.6) 100 (30.5) | 25 (7.6) 100 (30.5) |
| Mat Areal Basis Weight oz/square yard (gm/m ²) lb/3000 sq. ft. ream | 5.1 (173) 106 | 2.2 (75) 46 | 4.6 (156) 96 | 3.5 (119) 73 |
| MD/CD Tensile Ratio | 27.6 | 73.1 | 19.7 | 17.7 |
| MD Orientation of fibers, % | 96.5 | 98.7 | 95.2 | 94.7 |

- II. In one particular series of tests, multiple layers of the mat of example IB were stacked and molded under heat and pressure. The theoretical predicted composite modulus was calculated at 4.7 million psi (32.4 gigapascals). Measured modulus was 4.4 million psi. (30.3 gigapascals) which translates to 94% of theoretical.
- III. In another series of tests, an 8 inch (20cm) wide open headbox rotary cylinder wet forming machine was configured as in Figure 2A. The water chemistry system of Example 1 was used, with a viscosity of 3.5 centipoise. Wire velocity was 100 feet (30.5 meter) per minute, a 4/1 ratio to the 25 feet/minute (7.6 meter/minute) headbox stock velocity. Highly oriented products were made from the following materials:

Glass reinforcement fiber /PET, PAN Carbon/PET, and a hybrid reinforcement mixture of long (1.25 inch or 3.18cm) Glass with short 0.039 inches

(1 mm) Pitch Carbon Fibers. PET thermoplastic fibers were used.

Operating variables and resultant mat orientation ratios were as follows:

| Identification: | A | B | C | D | E |
|--|------------------------|------------------------|------------------------|----------------------|--|
| Reinforcing Fiber | Glass | Glass | Glass | PAN Carbon | 1) 47 wt. % Glass 2) 23 wt % Pitch Carbon |
| Reinf. Fiber Modulus Million PSI (gigapascals) | 10.5 (72.4) | 10.5 (72.4) | 10.5 (72.4) | 33 (22.8) | 1) 10.5 (72.4) 2) 82 (565) |
| Wt % Reinf. Fiber Vol. % Reinf. Fiber | 60 44 | 60 44 | 60 44 | 65 55 | 70 total 1) 36 2) 21 |
| Length, inches (cm) Reinf. Fiber | 1 (2.5) | 1 (2.5) | 1 (2.5) | 1.25 (3.18) | 1) 1.0 (2.5) 2) 0.039 (0.01) |
| PET Thermoplastic fiber | 0.5 (1.3) | 0.5 (1.3) | 0.5 (1.3) | 0.5 (1.3) | 0.5 (1.3) |
| Velocity feet/minute (meters/minute) Headbox Stock Forming Wire | 25 (7.6) 100 (30.5) | 25 (7.6) 100 (30.5) | 25 (7.6) 100 (30.5) | 25 (7.6) 200 (61) | 25 (7.6) 100 (30.5) |
| Mat Areal Basis Weight oz/square yard (gny/m ²) lb/3000 sq. ft. ream | 10.0 (339) 208 | 7.9 (268) 165 | 4.6 (156) 96 | 2.3 (78) 49 | 2.9 (98) 60 |
| MD/CD Tensile Ratio | 12.5 | 16.2 | 23.2 | 15.6 | 51.6 |
| MD Orientation of fibers, % | 92.6 | 94.2 | 95.9 | 93.9 | 98.1 |

- IV. In another series of tests, continuous fabrication of both flat and hat shaped beams was accomplished on the equipment disclosed in U.S. Patent 5,182,060, assigned to E.I. DuPont de Nemours and Co., herein incorporated by reference. These were laminated from stacks of mat with different compositions to demonstrate the concept of engineered hybrids. Flat beams were demonstrated up to 6 feet long (1.83 meters) and 4 inches (10cm) wide. Thickness measurements showed a final consolidation of 56%. Parts made consisted of:

- a) A single layer of the oriented pitch-based carbon mat of Example I-D on each surface, with eight layers of isotropic 0.5 inch (1.3cm) glass (25 wt%)/PET(75 wt%) in the center.
- b) A single layer of the oriented pitch-based carbon mat of Example I-D on each surface, with eight layers of the oriented 1.25 inch (3.18cm) glass /PET mat of Example I-B in the center.

V. In a different series of tests, the oriented carbon/glass hybrid of example IV-B was repeated with an additional layer of oriented carbon mat on one surface, and made into a 4 inch (10cm) wide "flat" beam. The resultant structure had a

natural radius of curvature in the direction of orientation (machine direction) of approximately 18 inches (46cm), with the double carbon layer surface toward the outside of the curve.

USES OF THE PRESENT INVENTION

5 As such, the present invention allows highly machine direction oriented large area fibrous mats to be produced at commercial speeds from the complete spectrum of natural and manmade fiber lengths, materials (including ceramics and metals), and compositions (mixtures of fiber materials and lengths), with or without thermoplastic components or other binders, on either of two major
10 classifications of wetlay machinery. Where headbox geometry is not suitable, the present invention utilizes principles which allow simple flow pattern modifications to attain high machine direction orientation, and temporary setup on many existing commercial machines. As such, the present invention readily lends itself to the retrofitting of existing machinery. Specific elimination of foam in wet
15 end processing minimizes floating fibers which tend to coalesce, tangle, and/or rope and diminish sheet quality and orientation. The mats are useful in high speed and/or automated production of reproducible structural parts and shapes. They can provide stiffness, reduced weight, strength, and engineered properties (physical, mass transfer, heat transfer, and electrical). In many applications, the
20 weight savings translate to significant energy savings.

When thermally or adhesively bonded, these mats yield high modulus, light weight, structural composites suitable for, but not limited to: automotive frames, other lightweight transportation (trucks, buses, trains, airplanes), infrastructure (commercial and home construction, column reinforcement,
25 acoustical materials), electronics (EMI, RFI shielding, cases, circuit boards, high strength insulators or conductors, heat sinks), membrane or filter reinforcements, heat sinks, consumer products including sporting goods, furniture frames, shoe parts, loudspeaker "horns", and many other applications requiring stiffness, and light weight. Laminated stacks may be of uniform composition, or of dissimilar
30 layers combined to produce engineered properties. Single or relatively few layers of mat may be used to stiffen and reinforce automotive headliners, thermal and acoustical insulation, etc. Both porous and fully consolidated structures may be produced. Materials such as films, foils, continuous fiber filaments or strands, or textile fabrics produced by woven, nonwoven, weft insertion, or knitting means,
35 may be inserted into the engineered stack, or onto it as decorative surfaces. Discrete patches of various shapes may be placed into or onto the stack automatically or by hand to provide desired localized properties. Oriented mats

- may be combined with mats of random, or other orientation. Products with controlled curvature may be produced by asymmetrically (from center of pile out), stacking layers of higher orientation, or higher stiffness (modulus). The porosity of the mat makes it suitable for stacking and efficient heating in a through - air
- 5 convection oven. The mat is also suitable for compression molding or hot stamping, continuous forming in a belt press, continuous shape forming by hot roller processing, continuous shape forming by reciprocal stamping (as disclosed in the aforementioned U.S. Patent No. 5,182,060), forming of shapes or rods by pultrusion, manufacturing structural shapes, and continuous manufacture of
- 10 structural rods, ropes, and cables.

Although the aforementioned embodiments have been shown and described in detail, it is to be understood that the scope of the invention is to be defined by the following claims.

CLAIMS

What is claimed is:

1. A method of producing highly-oriented fibrous mats having at least a 90% machine direction orientation using a wetlay machine having an open headbox and a moving wirebelt, said method comprising the steps of:
 - a) producing a thickened solution containing a plurality of suspended fibers, said thickened solution having a viscosity of equal to or greater than about 1.5 centipoise and said suspended fibers having fiber lengths of greater than about 0.6 cm and a modulus of at least 8 million psi;
 - b) introducing the thickened solution into a headbox of the wetlay machine and reducing its velocity to less than about 1/3 the velocity of said moving wirebelt; and
 - c) applying suction through said wirebelt to orient and pin said plurality of fibers on said wirebelt.
2. The method of claim 1 further comprising the step of adding an anti-foaming agent to said thickened solution.
3. The method of claim 1 further comprising the step of avoiding foaming agents within said thickened solution.
4. The method of claim 1 wherein said thickened solution is produced to have a constant viscosity under normal shear.
5. The method of claim 1 wherein said thickened solution is produced to have thixotropic properties.
6. The method of claim 1 wherein said thickened solution is thixotropic and produced to have a viscosity of at least 7 centipoise.
7. The method of claim 1 wherein said thickened solution contains a plurality of reinforcement fibers and thermoplastic components.

8. The method of claim 1 wherein said reinforcement fibers have fiber lengths in the range of about 0.6 cm to 6.35 cm.

5 9. The method of claim 9 wherein said reinforcement fibers have fiber lengths in the range of 1.9 cm to 3.2 cm.

10 10. The method of claim 7 wherein said reinforcement fibers have surface treatments designed to promote adhesion to said thermoplastic components.

11. The method of claim 7 wherein said reinforcement fibers are all made of one material and have at least substantially the same length and diameter.

12. The method of claim 7 wherein said reinforcement fibers are made of a mixture of materials, and have different lengths, diameters and compositions.

13. The method of claim 7 wherein concentration of reinforcement fiber components to thermoplastic components is in the range of 60-70% by weight of reinforcement fibers to 40-30% by weight of thermoplastic components.

14. The method of claim 7 wherein said thermoplastic component is selected from the group consisting of fibers, granular particles and flat platelets.

15. The method of claim 7 wherein said thermoplastic components are fibers with lengths in the range of 1/4" to 3/4" (0.6 to 1.9cm).

16. The method of claim 7 wherein said thermoplastic component is fibers selected from the group consisting of drawn and undrawn fibers.

17. The method of claim 7 wherein said thermoplastic components are made of the same material and are all substantially the same size.

18. The method of claim 7 wherein said thermoplastic components are made of a mixture of materials, and have different sizes and melting points.

19. The method of claim 7 further comprising the step of adding an additional material to the thermoplastic component selected from the group consisting of

fillers, antioxidants, coloring agents, electrically-conductive materials, electrically- insulating materials, thermally-conductive materials, thermally-insulating materials, adhesion aids, melt flow modifiers, cross-linking agents, chemically-reactive materials, biologically-reactive materials and molecular
 5 sieves.

20. The method of claim 1 further comprising the step of maintaining an open headbox.

10 21. The method of claim 1 further comprising the step of altering the internal shape of said headbox to eliminate deadspots containing eddy current formations.

22. The method of claim 21 wherein the step of altering the internal shape of said headbox comprises:

- 15 a) installing a plate extension in an upper portion of the headbox to deflect fibers and to prevent floating and entanglement of the fibers; and
 b) installing a regulating weir at an upper portion of the headbox downstream of said plate extension.

20 23. The method of claim 21 wherein the step of altering the internal shape of said headbox comprises installing a streamlined portion which conforms to a natural streamline flow of stock within said headbox.

24. Fibrous mats produced from the method of claim 1.

25

25. Structural composites produced from the method of claim 1.

26. The method of claim 1 wherein said thickened solution is introduced into the headbox uniformly across a width of the headbox and substantially vertically
 30 upward against a liquid head to slow and turn the plurality of fibers toward the wirebelt with reduced turbulence and with reduced linear velocity.

27. The method of claim 1 wherein said thickened solution is introduced into the headbox in a substantially backward and upward direction from the direction
 35 of the wirebelt, and is slowed against a liquid head to reverse flow of said plurality of fibers in a smooth pattern and to present the plurality of fibers to the wirebelt with reduced velocity and turbulence.

28. The method of claim 1 wherein a ratio of linear wirebelt velocity to linear thickened solution velocity is at least 3:1.

- 5 29. A method of retrofitting an existing headbox of a wetlay machine so as to produce highly-oriented fibrous mats, comprising the steps of:
- a) increasing head level within the headbox to increase headbox stock capacity; and
 - b) accelerating operating speed of a wirebelt within said wetlay machine
- 10 beyond an operating speed of stock in the body of the headbox.

30. The method of claim 29 further comprising the step of installing a plate extension in an upper portion of the headbox to deflect fibers and to prevent floating and entanglement of the fibers.

- 15 31. The method of claim 29 further comprising installing a regulating weir at an upper portion of the headbox and downstream of a last suction box in said headbox.

- 20 32. The method of claim 29 further comprising installing a curved portion within said headbox to eliminate dead spots and corresponding eddy current formations.

- 25 33. The method of claim 32 wherein said curved portion is a streamlined portion which conforms to a natural streamline flow of stock within said headbox.

- 30 34. The method of claim 29 wherein said step of increasing head level comprises increasing head level height so that the head level is vertically higher than a highest vertical position of a highest suction box disposed under said wirebelt within said headbox.

- 35 35. The method of claim 34 wherein the head level height is set at a height in the range of 18-26cm higher than an exit point of said highest suction box disposed under said wirebelt within said headbox.

36. A mat comprising a plurality of discontinuous reinforcement fibers, wherein said reinforcement fibers have at least a 9 to 1 machine to cross direction mat strength ratio.

- 5 37. A mat comprising a plurality of discontinuous reinforcement fibers having at least a 90% machine direction orientation.

38. The mat of claim 37 wherein a basis weight of said mat falls within the range of 68 to 339 gm/square meters.

10

39. The mat of claim 37 wherein said mat further contains a thermoplastic component.

40. A product comprising a plurality of mats, each of said mats comprising a
15 plurality of discontinuous reinforcement fibers having at least a 90% machine direction orientation.

41. The product of claim 40 wherein at least one of said mats has been heated in an oven, compression molded, hot stamped, continuously formed in a belt
20 press, continuously shape-formed by hot roller pressing, continuously shaped by reciprocal stamping, formed through pultrusion, or continuously manufactured to form structural rods, ropes and cables.

42. The product of claim 40, wherein each of said mats have different fiber
25 components and fiber orientations.

Title**WETLAY PROCESS FOR MANUFACTURE OF
HIGHLY-ORIENTED FIBROUS MATS****ABSTRACT**

A mat containing highly machine direction oriented (90% or greater), discontinuous reinforcement fibers, is produced on inclined wire or rotary paper making machinery. Fibers are first uniformly dispersed in an aqueous medium containing thickeners and wetting agents. In one embodiment, antifoaming agents are also added to prevent floating fibers which entangle and reduce orientation. Thermoplastic fibers or particles may also be included. Stock is brought into an open headbox in a flow pattern which allows the fibers to decelerate before approaching the porous suction belt (wire). As the fibers approach the suction belt, the fibers begin to turn and align in the streamline so as to present one end toward the suction wire. The leading ends of the fibers are gripped by the moving belt which drags the fibers out of the dispersion stock in a straight line. The porous mat produced may be dried and bonded through hot air, heat and/or pressure, or chemical binders. Stacks of such mats may be compressed partially to produce porous structures, or fully to produce impervious, rigid structural panels or shapes.

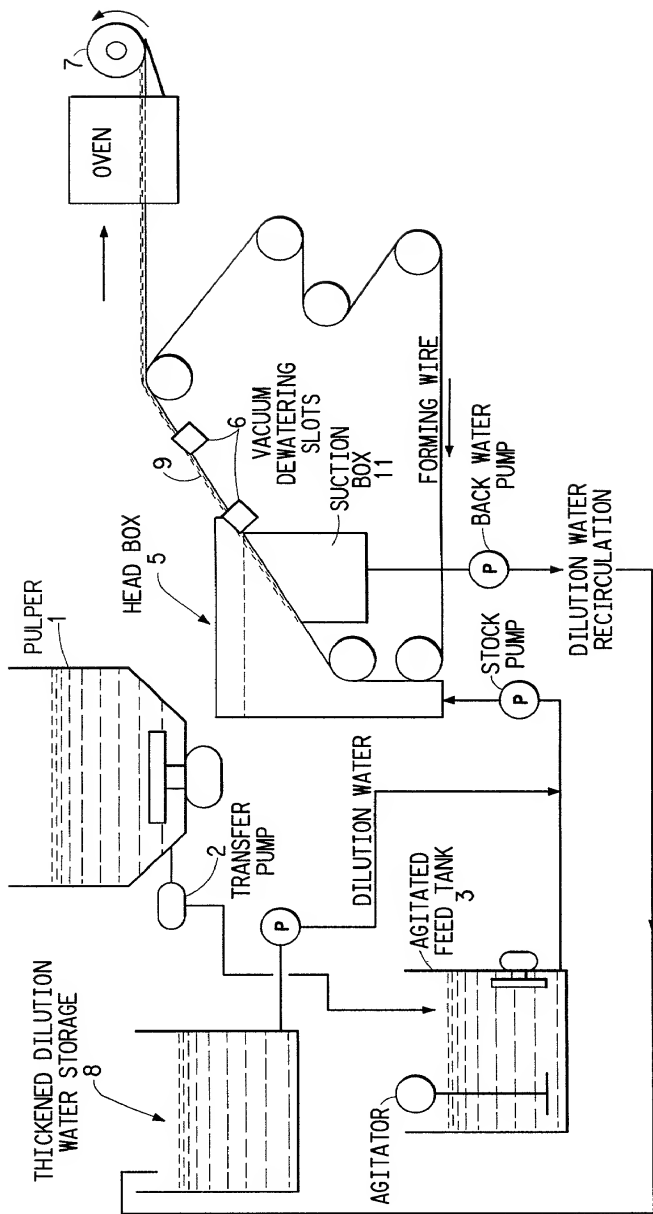


FIG.1

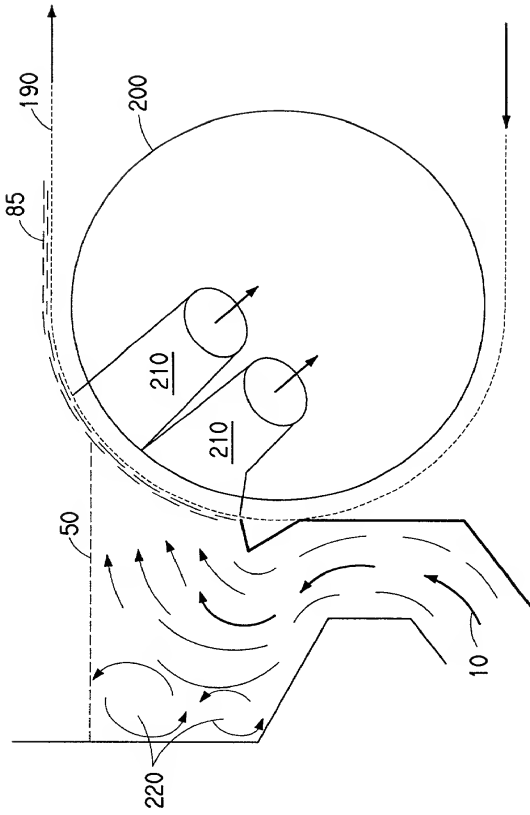


FIG. 3A

DECLARATION and POWER OF ATTORNEY

As a below-named inventor, I hereby declare that:

My residence, post office address and citizenship are as stated below next to my name.

I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled:

WETLAY PROCESS FOR MANUFACTURE OF HIGHLY ORIENTED FIBROUS MATS

the specification of which is attached hereto unless the following box is checked:

☐ was filed on _____ as U.S. Application No. _____ or PCT International Application No. _____ and was amended on _____ (if applicable).

I hereby state that I have reviewed and understand the contents of the above identified specification, including the claims, as amended by any amendment referred to above.

I acknowledge the duty to disclose information which is known to me to be material to patentability as defined in 37 CFR § 1.56.

I hereby claim foreign priority benefits under 35 U.S.C. § 119(a)-(d) or § 365(b) of any foreign application(s) for patent or inventor's certificate, or § 365(a) of any PCT International application which designated at least one country other than the United States, listed below and have also identified below, by checking the box, any foreign application for patent or inventor's certificate, or PCT International application having a filing date before that of the application on which priority is claimed.

| Application No. | Country | Filing Date | Priority Claimed (Yes/No) |
|-----------------|---------|-------------|---------------------------|
|-----------------|---------|-------------|---------------------------|

I hereby claim the benefit under 35 U.S.C. § 119(e) of any United States Provisional Application(s) listed below.

U.S. Provisional Application No.

U.S. Filing Date

I hereby claim the benefit under 35 U.S.C. § 120 of any United States application(s), or § 365(c) of any PCT International Application designating the United States, listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States application or PCT International Application in the manner provided by the first paragraph of 35 U.S.C. § 112, I acknowledge the duty to disclose information which is known to me to be material to patentability as defined in 37 CFR § 1.56 which became available between the filing date of the prior application and the national or PCT International filing date of this application.

| Application No. | Filing Date | Status (patented, pending or abandoned) |
|-----------------|-------------|---|
|-----------------|-------------|---|

POWER OF ATTORNEY: I hereby appoint the following attorney(s) and/or agent(s) the power to prosecute this application and transact all business in the Patent and Trademark Office connected therewith:

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HANH T. PHAM

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

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☐ Additional Inventors are being named on separately numbered sheets attached hereto.